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Twenty-fourth day of November 2003

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Patents Act 1990

PROVISIONAL SPECIFICATION

Applicant(s):

COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION

Invention Title:

MERCERISATION OF CELLULOSIC FIBRES

The invention is described in the following statement:

MERCERISATION OF CELLULOSIC FIBRES

TECHNICAL FIELD

The invention relates to a process and apparatus for the mercerisation of unconstrained cellulosic fibres.

DESCRIPTION OF PRIOR ART

The beneficial effects obtained by treating cotton with concentrated solutions of various reagents were described by John Mercer in British Patent 13,296 dated 1850. In particular, he noted that treatment of cotton fabrics with sodium or potassium hydroxides, followed by thorough rinsing, produced shrinkage and thickening similar to that obtained when woollen fabrics are milled. He also noted an increase in tensile strength and improved affinity for dyestuffs following the treatment.

Whilst the original process developed by Mercer improved the strength of the cotton fabric, the fabric became thicker and more dense due to shrinkage. The "handle" or lustre of the fabric was also deteriorated as a consequence of the treatment.

It was not until 1890 that the process now known as mercerising was first described by Lowe in British Patent 4452. Mercerising is a variation on the original process developed by Mercer under which a cotton fabric or yarn is treated with the mercerising reagent (usually sodium hydroxide) in a stretched condition, followed by rinsing to remove the reagent. Lowe found that shrinkage of the cotton cloth or yarn could be prevented by maintaining tension in the cotton cloth or yarn during the treatment and rinsing steps. Another important effect of maintaining tension on the material during removal of the

alkali was the development of a very high degree of lustre. Other benefits, previously found by Mercer, such as increased strength and improved affinity for dyes, were also obtained by the method carried out under tension.

The mercerisation process, as is now most commonly practised, involves immersing cotton in a concentrated, aqueous solution of sodium hydroxide (or less commonly another mercerising liquid), followed by rinsing with water and neutralising any alkali remaining in the rinsed cotton with dilute acid. As discussed above, the process is carried out under tension. The powerful swelling action of sodium hydroxide (or other mercerising liquid) has a marked effect on the structure and properties of cellulose. The convolutions present in untreated cotton fibres are removed and their shape is changed from a flat ribbon-like structure to a smoother more cylindrical form. The effect of the mercerising liquid in swelling the cellulose is that the central cavity (lumen) present in raw cotton largely disappears and the fibre material tends to fill the whole cross-section. Native cotton contains both crystalline and amorphous cellulose and mercerising increases the proportion of amorphous material in the fibre. Mercerisation also improves the following properties of cotton:

- Colour yield for a given dye concentration.
- Dye coverage of immature fibres.
- Chemical reactivity.
- Tensile strength.
- Strength retention following application of easy-care finishes.
- Lustre.

Cotton fibres are roughly one inch long, although the fibre length can range from less than half an inch in length to around 2 inches. Until the fibres are aligned

and spun into yarn form, assemblages of the fibres, such as carded cotton, slivers and rovings, do not have the ability to be stretched by pulling either end of the assemblage. For example, if either end of a sliver of cotton is pulled, the cotton fibres will slide over one another, and will not individually be stretched. In contrast, cotton yarn and fabric can be stretched at either end of a length thereof to effect stretching of the individual fibres. As a consequence, cotton is mercerised in fabric or yarn form.

Procedures for mercerising cotton fibres or sliver, which prevent fibre shrinkage, are not commercially available, because of the above-described practical difficulties of maintaining tension on an unconstrained assembly of fibres during the mercerising process. An industrial procedure for mercerising unconstrained cellulosic fibre such as cotton would be of significant commercial benefit, because in addition to the improvements listed above, the following benefits could also be anticipated:

- Improved evenness of treatment resulting from subsequent processing carried out after mercerising the fibre.
- Increased fibre strength, compared with normal cotton. This would lead to improvements in spinning performance, in particular the possibility of higher spinning frame speeds.
- Possibility of blending mercerised cotton fibre with alkali-sensitive materials, in particular keratin fibres such as wool.
- Knitted fabrics can be made from mercerised cotton. Although knitted fabrics can be mercerised, the mercerisation process tends to give a decrease in fabric bulk. This problem would be avoided if cotton could be mercerised as loose fibre or sliver.

Whilst there has been interest in an industrial treatment for mercerising unconstrained cotton fibres under tension for many years, no satisfactory continuous process has been developed. Accordingly, it is an object of the present invention to provide a practical process for mercerising unconstrained cellulosic fibres. Preferably, the process should be capable of being conducted on a continuous basis, and should provide the full benefits of tension mercerisation, including retention of fibre length and development of lustre.

BRIEF SUMMARY OF THE INVENTION

The present invention provides a process for mercerising unconstrained cellulosic fibres comprising:

transporting the unconstrained cellulosic fibres along a transit path through a mercerising zone, in which the unconstrained cellulosic fibres are contacted with a mercerising liquid followed by a rinsing zone, in which the unconstrained cellulosic fibres are rinsed;

wherein the unconstrained cellulosic fibres are held to prevent longitudinal shrinkage during transportation through both zones.

The term "unconstrained" cellulosic fibres as used in this specification refers to oriented or disoriented cellulosic fibres which have not been subjected to spinning or twisting to the extent that they form a yarn. Accordingly, this term encompasses raw fibres, carded fibres, slivers and rovings, and excludes yarns and fabrics. The cellulosic fibres are suitably in the form of a sliver.

The term "cellulosic fibres" has been used in this specification in its broadest sense, and includes cotton fibres, linen fibres, viscose and combinations

thereof.

The fibres should remain held throughout each of the mercerising and rinsing steps and the passage between these two steps. If the fibres are not held to prevent shrinkage throughout the passage following the mercerising step leading up to the rinsing step, then the fibres will shrink on release of the holding force. In order to regain the original fibre length it would be necessary to re-stretch the fibres. For this reason, it is necessary for the fibres to be held to retain the fibre length during the entire sequence of the mercerising step followed by the rinsing step, unless the fibres are re-stretched before rinsing. Once most of the mercerising liquid has been removed in the rinsing step, then the holding or tension can be released, without penalty, for example in an acid neutralising step following the rinsing step.

Preferably, the unconstrained cellulosic fibres are held by being compressed between two surfaces as they travel along the transit path. Indeed, it is preferred that the cellulosic fibres be transported along the transit path between a pair of belts which are pressed together in a plurality of positions through the transit path. The belt should be under tension to maintain a compression force on the fibres to hold them firmly and thus prevent longitudinal shrinkage.

It has been found by the present applicant that excellent results are obtained when the fibres are stretched (placed under stretching tension) during transportation through the mercerising and rinsing zones. Stretching of fibres in the two zones can be achieved by using a slightly elastic belt, which is placed under tension through the mercerising and rinsing zones. Since this arrangement will stretch the fibres in the direction of movement of the belt, it is preferred that the

unconstrained cellulosic fibres be oriented. Preferably the unconstrained cellulosic fibres are in the form of carded cotton, a sliver or a roving.

The belts may be made of any suitable material that is resistant to the action of the mercerising liquid. Various polymeric materials are particularly suitable for this purpose, such as polyester.

Preferably, the transit path includes nip rollers at the beginning and end of the transit path, which place the belts under tension through the two zones therebetween. The oriented unconstrained cellulosic fibres may be fed into the process between the two belts at the first of the nip rollers. As a consequence of this, if the belts are under tension and are slightly elastic, the fibres are slightly stretched with the belt as they are fed into the process.

Preferably the belts pass over guide means at a number of points along the transit path. It has been found by the applicant that for optimum efficiency in preventing slippage of the fibres between the belts, the straight path length between the points at which the belt is in contact with a guide means should be in the range of the average length of the cellulosic fibres or less than this distance. For example, for cotton fibres having an average length of around one inch (2.5 centimetres), the straight path length should be less than 2.8 cm, and preferably 2.5 cm or less.

Preferably, the guide means are rollers. The diameter of the rollers is of less significance than the path length between the rollers, however rollers of small diameter are preferred. The diameter of the rollers may advantageously be less than two times the average length of the fibres being treated. More preferably, the

diameter of the rollers is less than 1.5 times the average length of the fibres being treated, and most preferably less than or equal to the average length of the fibres.

Preferably, the transit path is a circuitous path that winds around the rollers. More preferably, there are approximately equal numbers of left- and right-hand curves around the rollers in the transit path.

Further nip rollers in addition to those at the beginning and the end of the transit path may be provided. For example, nip rollers may be provided in the region between the mercerising zone and the rinsing zone to squeeze excess mercerising liquid out of the fibres and belts.

The belts may be driven by any appropriate means. For example, the nip roller at the end of the transit path may be configured as a pad mangle which pulls the belt through the transit path.

The mercerising liquid is preferably contained in a mercerising bath, and the rinse liquid in a rinse bath.

Any suitable mercerising liquid may be used in the process of the present invention. However, it is preferred that the mercerising liquid is an alkali. Preferably further, the alkali is a concentrated solution of sodium hydroxide or potassium hydroxide. Appropriate concentrations for the alkali are known in the art. Preferably the concentration of the alkali is between 15 and 30% mass volume (grams per 100 ml), or 35 -55°Tw (Degrees Twaddell - a measure of specific gravity used in the art of the invention). More preferably, the concentration is about 20-22% or about 40 °Tw. Alternative mercerising liquids may include strong acids (eg sulphuric or phosphoric) zinc chloride, calcium chloride

and so forth.

Preferably, the mercerising liquid also contains a wetting agent. Suitable wetting agents (ie ones that are stable in concentrated solutions of alkali) are known in the art.

Preferably, the rinsing liquid is water, however the rinsing liquid may be any other liquid having a pH less than that of the mercerising solution, in the case where the mercerising liquid is an alkali.

The cellulosic fibres may be transported through additional zones after rinsing. For example, the cellulosic fibres may be conveyed through a neutralising zone in which the cellulosic fibres are neutralised with a neutralising liquid. The neutralising liquid may for example be a dilute acid.

Preferably, the process increases the lustre of the fibres by at least 50% compared with the original fibres prior to mercerising, as measured by the amount of reflected light obtained in a test in which light is shone onto the fibres at a 90° angle and reflected light is measured at a 45° angle. More preferably, the increase in lustre of the fibres is increased by at least 100% and most preferably at least 150% as measured by this test.

According to the present invention, there is also provided an apparatus for mercerising unconstrained cellulosic fibres, the apparatus comprising:

(i) a mercerising zone;

(ii) a rinsing zone following the mercerising zone;

(iii) a conveyor comprising a pair of surfaces for holding the unconstrained cellulosic fibres to prevent longitudinal shrinkage during transportation of the unconstrained cellulosic

fibres along a transit path through the mercerising zone and the rinsing zone;

(iv) driving means for driving the conveyor; and

(v) pressure means for pressing the surfaces of the conveyor together in the mercerising zone and the rinsing zone to thereby hold the unconstrained cellulosic fibres so as to prevent longitudinal shrinkage of the fibres through the zones.

Preferably, the apparatus also includes a neutralising zone following the rinsing zone.

Preferably, the conveyor is configured to enable the unconstrained cellulosic fibres to be held to prevent longitudinal shrinkage throughout the mercerising zone, the rinsing zone and the passage between these two zones. It is not, however, required for the fibres to be held during transportation through the neutralising zone.

Preferably, the conveyor comprises a pair of belts. Preferably the belts are under tension so that, in use, a compression force is maintained on the fibres to hold them firmly and thus prevent longitudinal shrinkage. For the reasons explained above, preferably the belt is slightly elastic so that, in use, an assembly of aligned, unconstrained cellulosic fibres fed between the belts are subjected to a stretching force by the belts in the mercerising and rinsing zones.

Preferably, the pressure means includes at least two pairs of nip rollers, one pair of nip rollers being located at the end of the mercerising zone, to squeeze out excess mercerising liquor, and the other pair being located at the end of or following the end of the rinsing zone. The pairs of nip rollers should place the belts under tension in the mercerising and rinsing zones therebetween.

Preferably, the straight distance between the points at which the belts come into contact with pressure means ("path length") along the transit path is in the range of the average length of the cellulosic fibres to be treated in the apparatus, or less than this distance. Preferably, the path length is less than 2.8 cm, more preferably 2.5 cm or less.

Preferably, the pressure means includes a plurality of rollers located along the transit path in the mercerising zone and the rinsing zone. Preferably, the diameter of the rollers is less than two times the average length of the fibres to be treated in the apparatus, more preferably less than 1.5 times, and most preferably less than or equal to the average length of the fibres.

Preferably, the transit path is a circuitous path that winds around the rollers, and there are approximately equal numbers of left- and right-hand curves around the rollers in the transit path.

Preferably, the drive means is in the form of a mangle at or downstream of the end of the transit path. The drive means may be constituted by the final pair of nip rollers at the end of the transit path which have the dual effect of squeezing out of the fibres.

The apparatus may include further components, such as further nip rollers to control the transport of the belt through the mercerising liquid and the rinsing and neutralising liquids.

Preferably, the mercerising zone includes a mercerising bath for containing a mercerising liquid, the rinsing zone includes a rinsing bath for containing a rinsing liquid, and the neutralising zone (if present) includes a neutralising bath for containing a neutralising

liquid. The neutralising liquid may for example be a dilute acid.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention is described further by way of example with reference to the accompanying figure in which:

Figure 1 is a schematic diagram of the mercerising apparatus in accordance with a preferred embodiment of the invention; and

Figure 2 contains scanning electron micrographs of cross-sections of cotton fibres before and after treatment in the apparatus of Figure 1.

DETAILED DESCRIPTION OF THE INVENTION

Figure 1 schematically illustrates apparatus according to a preferred embodiment of the invention.

The apparatus for mercerising unconstrained cellulosic fibres according to the preferred embodiment as illustrated in Figure 1 includes a first bath 10 containing a mercerising liquid, a second bath 12 containing a rinse liquid, a pair of enveloping conveying belts, including an upper belt 14 having a return loop passing over the baths, and a lower belt 16 having a return loop passing beneath the baths, and a series of rollers including nip rollers 18, 20, 22 and guide rollers 24a-k for applying a pressing force to press the belts together between rollers 18 and 22. Nip rollers 22 are constituted as a pad mangle which drives the belts in the direction indicated by the arrows.

The belts 14,16 are formed from a material that is slightly elastic and resistant to the mercerising

liquid in the first bath 10. The construction of the belts allows the mercerising liquid used in the process to penetrate into the sliver, and also allows the rinsing liquid to fully rinse the sliver. The belts are driven through a transit path which extends from nip roller 18 to nip roller 22. The transit path of the belts runs through the first bath 10, out of the first bath and through the second bath 12 in the direction of travel of the belts 14,16.

The transit path of the belts is circuitous and runs around a series of closely spaced rollers 20 and 24a-k of small diameter. The diameter of the rollers of the preferred embodiment illustrated is 5 cm. This is appropriate for cellulosic fibres of an average length of about 2.5 cm. The diameter of the rollers may be adjusted. The straight path length of the belt between adjacent rollers is measured between the points where it comes into contact with adjacent rollers. The straight path length is approximate to or less than the average fibre length (in this case 2.5 cm). In the preferred embodiment illustrated, the straight path length between adjacent rollers ranges from 2.8 cm to 0 (in the case of the nip rollers). The distance between the rollers may also be adjusted.

The rollers are arranged so that the belt follows a meandering path through the mercerising bath and the rinsing bath, with an approximately equal number of left- and right-hand curves.

Unconstrained cellulosic fibres, preferably in the form of a sliver 26, are fed between the two belts from nip roller 18 to nip roller 22. The fibres in the sliver are aligned to the direction of travel of the belts and the direction of feeding of the sliver into the apparatus (arrow A).

The pad mangle 22 pulls the belt along the transit path and applies tension to the belt between the pad mangle and nip roller 18. As the belt is slightly elastic, the belt is stretched commencing at nip rollers 18. The input sliver 26 that is fed between the belts at nip rollers 18 is compressed between the belts to be held sufficiently firmly to prevent shrinkage of the fibres as they pass through the transit path. Since the belt is slightly stretched as it commences the transit path at nip rollers 18, so too are the fibres between the belts. The degree of stretching of the fibres is around 1-2%. The fibres remain in the stretched state as they travel along the transit path through the mercerising bath, out of the mercerising bath and through the rinsing bath. The tension applied to the fibres also assists to maintain the fibres parallel to the direction of travel through the apparatus.

As explained previously, the small diameter of the rollers 20 and 24a-k and the short path length between the rollers (relative to the fibre length) has been found to provide optimum efficiency in preventing slippage of the fibres between the facing surfaces of the pair of belts 14,16.

Nip rollers 20 are positioned above the mercerising bath and squeeze excess mercerising liquid out of the sliver and belts. The excess mercerising liquid drains back into the bath.

In the preferred embodiment of the invention, the mercerising liquid is an aqueous solution of sodium hydroxide. The concentration of the sodium hydroxide is 20-22% mass volume or 40°Tw at 15°C, which corresponds to a specific gravity of 1.2 or approximately 21 grams of sodium hydroxide per 100 ml of water. The mercerising

liquid also contains a wetting agent in the amount of 5 g/L to 25 g/L. One of the wetting agents which is stable in strong alkali known in the art is used in the preferred embodiment.

The mercerising solution is maintained at a temperature in the range of -5°C to 25°C.

The rinsing liquid has a pH less than the pH of the mercerising solution. Water is used in the preferred embodiment.

The sliver 26 may also be transported through additional zones (not shown) to expose the sliver to further solutions. An additional zone may include a solution containing a dilute acid to further neutralise any remaining mercerising liquid in the sliver and/or belts.

Whilst baths are used for containing the mercerising liquid and rinsing liquid in the preferred embodiment illustrated, it will be appreciated to a person skilled in the art of the invention that a chamber or chambers in which the unconstrained cellulosic fibres are sprayed with the liquids could be used in place of the baths. The term "zone" should accordingly be interpreted broadly to encompass this.

The speed at which the sliver 26 is transported through the mercerising bath 10 (and the length of the bath) should be sufficient to ensure that the mercerising liquid will have fully penetrated the sliver. This also applies to the other baths through which the sliver is conveyed.

Although the cellulosic fibres are held in a compressed condition between the tensioned belts 14,16, it

has been surprisingly found by the present applicants that the fibres are still able to swell and expand in diameter. Consequently, the fibres are changed by the mercerisation process from a flat, ribbon-like shape that is typical of untreated cellulosic fibres to the more circular cross-section that is characteristic of mercerised cellulosic fibres.

EXAMPLE

A 5 ktex sliver, composed of cotton fibres, was treated in the apparatus shown in Figure 1. The sliver was fed between two belts into a pair of nip rollers. It was carried between the belts around a series of rollers immersed in a tank containing the mercerising liquor, which consisted of an aqueous solution of sodium hydroxide at a concentration of 21.4% (mass/volume), maintained at a temperature below 10°C. The treatment liquor also contained 7g/L of a wetting agent (Leophen MC [BASF]). The speed of the belt and sliver was set to give an immersion time in the sodium hydroxide solution of 40 seconds. After squeezing out the excess sodium hydroxide solution, the sliver, while still held under tension, was then passed through a rinse bath, containing water, for 30 seconds. After rinsing, excess liquid was removed with a pair of squeeze rollers (not shown in Figure 1). The sliver was then passed into a bath containing dilute acetic acid, maintained at pH 5 (not shown in Figure 1), where the remaining alkali was neutralised.

A control was prepared by treating a cotton sliver with the solution of sodium hydroxide and wetting agent, described above; but in this case, the material was allowed to fully relax in the treatment solution and during the rinsing and neutralisation steps (this sample is referred to as slack mercerised).

Examination of the slivers after drying showed that the sample mercerised under tension on the Belt Mercerising Machine was more lustrous than the untreated material and also more lustrous than the slack mercerised sample. The lustre was measured as a function of the amount of reflected light obtained in a test in which light is shone onto the fibres at a 90° angle and reflected light is measured at a 45° angle. The lustre of the sliver was twice that of the untreated material. The fibre length, strength and elongation at break of the untreated and treated slivers were measured. The data in Table 1 show that the slack mercerised fibres contracted in length, whereas the fibres mercerised under tension were slightly longer than the untreated material. Both the slack mercerised and belt mercerised fibres were stronger and had a higher elongation at break than the untreated cotton.

Table 1:
Physical Properties of Untreated, Slack Mercerised and Belt (Tension)
Mercerised Cotton

	Mean fibre length (inches)	Fibre Strength (grams force per tex) (a)	Elongation at Break (%)
Untreated	1.17	32.5	6.0
Slack Mercerised	1.12 (- 4.3 %)	42.3 (+ 30.2%)	13.3
Belt Mercerised (under tension)	1.18 (+ 0.9 %)	41.5 (+ 27.7%)	8.9

The figures in brackets show percentage change compared with untreated cotton,

(a) gauge length 1/8 inch

Scanning electron micrographs of untreated and belt mercerised cotton (Figure 2) show that the belt mercerising technique successfully changed the fibre cross-section from the kidney-shaped, ribbon-like cross-section of normal cotton fibres to the more circular shape characteristic of mercerised cotton.

The above preferred embodiment and example are provided by way of illustration of the inventive concept only. Various modifications may be made to the preferred embodiment and example without departing from the spirit and scope of the invention.

Dated this 2nd day of December 2002
COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION
By their Patent Attorneys
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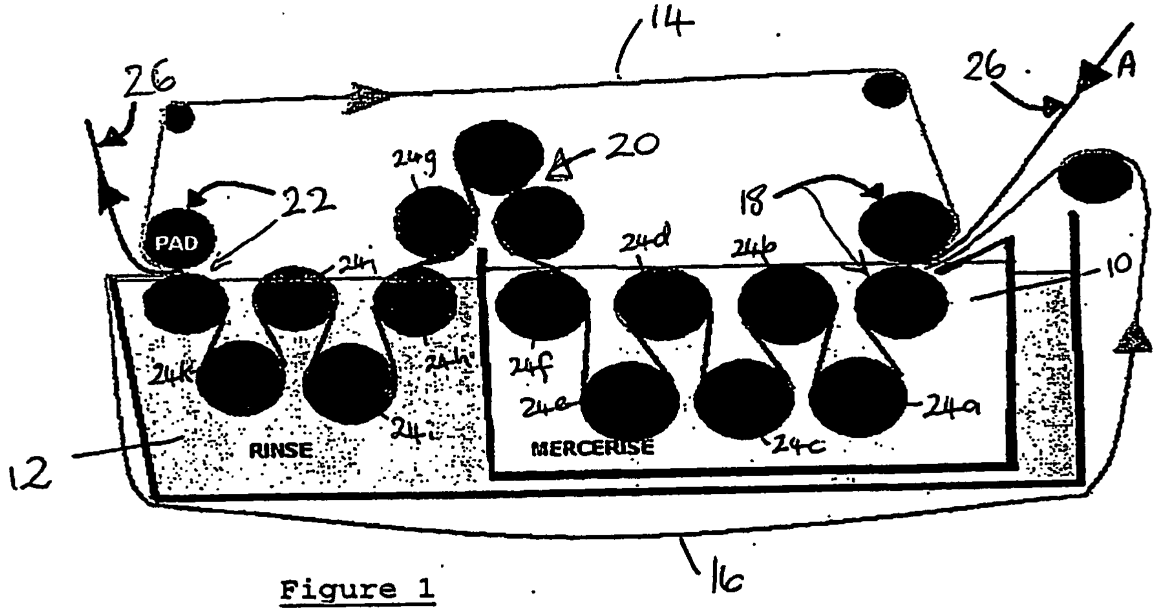

Figure 1

Untreated

Belt Mercerised

Figure 2